



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/EP99/00497 <b>(22) International Filing Date:</b> 26 January 1999 (26.01.99) <b>(30) Priority Data:</b> 9804283.1 27 February 1998 (27.02.98) GB <b>(71) Applicant (for AU BB CA CY GB GD GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZW only):</b> UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). <b>(71) Applicant (for all designated States except AU BB CA CY GB GD GH GM IE IL IN KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZW):</b> UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 Al Rotterdam (NL). <b>(71) Applicant (for IN only):</b> HINDUSTAN LEVER LIMITED [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Mumbai 400 020 (IN). <b>(72) Inventor:</b> MOHAMMADI, Mansur, Sultan; Unilever Research Port Sunlight, Quarry Road East, Bebington, Wirral, Merseyside L63 3JW (GB).		<b>(74) Agent:</b> MOLE, Peter, Geoffrey; Unilever PLC, Patent Dept., Colworth House, Sharnbrook, Bedford MK44 1LQ (GB). <b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> FABRIC CONDITIONING CONCENTRATE <b>(57) Abstract</b> <p>The invention relates to fabric conditioning concentrate compositions comprising a fabric softening compounds mixed with oil. In use, the fabric softening concentrate is diluted with water. In order to improve the viscosity, stability and fabric softening quality provided by the emulsion, a water-soluble polymeric structurant is used. The water-soluble polymeric structurant can be suspended in the conditioning concentrate itself or it can be first of all dissolved to the final desired concentration in the water used to dilute the fabric softening concentrate.</p>		

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## Fabric Conditioning Concentrate

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### Technical Field

The present invention relates to fabric conditioning concentrates with less than 10% by weight of water that easily disperse and self-emulsify on dilution with cold or warm water (10-70°C) and when emulsified in water give stable emulsions with controlled viscosity and creaming and which have excellent perfume delivery and softening for laundry.

### 15 Background and Prior Art

Conventional rinse conditioners are obtained by dispersing a cationic softening material and perfume into hot water. The problem with such conventional aqueous rinse conditioners is that although the rinse conditioners soften laundry they do not deliver perfume onto the fabric well because as much as one third of the perfume in the formulation remains in the rinse water. The dilute versions of such conventional aqueous fabric rinse conditioner may typically have a relatively low viscosity making them less attractive for use by consumers. Attempts have been made in the past to thicken such fabric softening compositions using thickener such as cationic starch as disclosed in EP-A-0596580 in the name of Avebe. However, it is found that it is very difficult to

achieve consistent and desirable results. In particular, cationic fabric softeners are typically added to aqueous compositions as concentrated aqueous solutions and viscosity control is difficult.

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European patent application no. 97306076.7 discloses a conditioning concentrate comprising a cationic fabric softening compound and oil in which the cationic fabric softening compound is suspended in the oil. When the concentrate is mixed with water, an oil-in-water emulsion is produced.

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GB 2 007 734 (Cargo Fleet) discloses a liquid fabric softener concentrate which consists of a quaternary ammonium salt in a dissolved state having at least one C<sub>8</sub>-C<sub>30</sub> long chain alkyl group and an oil. The concentrate can be dispersed or emulsified with water. No mention of improved perfume delivery is made.

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We have found that such emulsion-based softeners perfume fabrics more effectively than conventional aqueous rinse conditioners but subsequently have a loss in their softening performance.

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The concentrate compositions of the prior art are typically used by dosing the concentrate directly into the washing machine so that it is diluted with cold water during the rinse cycle. It has been found that if emulsions are produced using the concentrate compositions of the prior art, the emulsions are sometimes not stable on storage under

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ambient conditions (temperatures of 0-45°C). The emulsions are found to be particularly unstable at relatively high ambient temperatures or at relatively low concentrations of fabric conditioner (approximately 5% by weight). Further, emulsions produced using prior art concentrates tend to have relatively low viscosities or viscosities which vary from batch to batch, making them unattractive for the consumer to use.

10 EP-A-0730023 discloses laundry concentrate compositions comprising a mixture of at least one non-ionic surfactant and at least one water-insoluble fatty oil containing a hydrophilic polar group and having a melting point below 30°C, wherein the concentrate has viscosity in excess of  
15 about 10 mPas. Upon dilution with at least about one volume of water per volume of concentrate, the concentrate is converted at least partially into a liquid crystal phase dispersion, providing a diluted concentrate having a viscosity of at least about 50 mPas.

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It has been found that it is difficult to achieve the same softening effect with prior art concentrates as with conventional aqueous dispersions of oil based fabric softening components. It has been found that prior art  
25 fabric conditioning concentrates can impart a greasy feeling to the fabric. It is desired to achieve a silky feeling to the conditioned fabric.

We have now discovered that water-soluble polymeric  
30 structurant can be used to overcome the problems of the

prior art. The water-soluble polymeric structurant can be included in a fabric conditioning concentrate composition comprising fabric softener and oil. This fabric conditioner concentrate can be diluted with water to produce an emulsion. In another aspect, the water-soluble polymeric structurant can be dissolved in water to the final desired concentration and the solution of water-soluble structurant then used to dilute a fabric conditioning concentrate composition comprising a fabric softening compound and oil.

The use of water-soluble polymeric structurant overcomes the problems associated with the prior art. In particular, it can provide a fabric conditioning concentrate which can be diluted to give a stable, dilute emulsion (have concentrations of cationic fabric softener in the region 1-15%, preferably around 5% by weight). Such emulsions can have viscosity which is relatively consistent from batch to batch and in the desired range (being in the range 10-100 mPa.s at shear rate of  $106 \text{ s}^{-1}$ ). Further, the emulsions produced using the rinse conditioner concentrates of the present invention have excellent perfume delivery and further improved fabric softening properties. The present invention has overcome the undesirable greasy softness feeling that oil-based conditioners impart to fabrics, achieving instead a silky softened feeling.

Additionally the present invention can provide fabric conditioning concentrates which are not detrimental to the

absorbency of fabric and which also reduce the creasing of fabric. Fabric softened with the compositions of the present invention show better ease of ironing than conventional fabric softeners.

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#### Definition of the Invention

The present invention provides a fabric conditioning concentrate composition comprising a fabric softening compound mixed with oil, wherein the composition contains  
10 a water-soluble polymeric structurant.

The present invention further provides a method of manufacturing a fabric conditioning concentrate  
15 composition as described above comprising mixing together a fabric softening compound, oil and a water-soluble polymeric structurant.

The present invention also provides a method of preparing  
20 an aqueous emulsion containing a fabric conditioning compound, comprising diluting the fabric conditioning concentrate of the invention with water. The method can for example be carried out in a batch process at a factory or in a bottle at home.

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The present invention further provides a method of preparing an aqueous emulsion containing a fabric conditioning compound, comprising dissolving a water-soluble polymeric structurant in water to provide a  
30 solution of polymeric structurant of a concentration

substantially equal to the desired final concentration of water-soluble polymeric structurant in the emulsion, using the solution of polymeric structurant to dilute a fabric conditioning concentrate composition comprising a fabric softening compound mixed with oil. Preferably, this method is carried out as a batch process.

The present invention further provides a fabric conditioning composition comprising an aqueous emulsion, the aqueous emulsion comprising a fabric softening compound, oil and a water-soluble polymeric structurant.

The conditioning concentrate and methods of the present invention are distinguished from the prior art in that the water-soluble structurant is dissolved to its final desired concentration before or simultaneously with the production of an emulsion of fabric softening component in water. By mixing the water soluble polymeric structurant component with oil, it becomes possible to disperse in water the fabric softening compound and oil with little loss of the structure provided in the emulsion by the polymeric structurant.

#### Detailed Description of the Invention

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The term "concentrate" in the context of the present invention means that little or no water is present in the formulation. The maximum level of water that can be present in the formulation is 10% or less by weight of the total formulation, more preferably 5% or less by weight, most

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preferably 2% or less by weight. In some situations less than 0.5% by weight of water may be present.

A structurant is here defined as a material which  
5 increases the viscosity of an aqueous emulsion of the present invention or an aqueous emulsion formed by diluting the concentrate of the invention in water, both at gravitational or zero shear rate and at normal pouring shear rates.

10 Normal pouring shear rates are in the range 10 to about  $110 \text{ s}^{-1}$ .

Desirably, the viscosity of an emulsion according to  
15 the present invention is in the range 40-80 mPa.s at  $106 \text{ s}^{-1}$ , preferably around 60 mPa.s at  $106 \text{ s}^{-1}$ . It is found that little or no phase separation occurs under these conditions.

20 Without wishing to be bound by theory, it is believed that the increase in the zero shear rate viscosity of the composition provides kinetic stabilisation against gravitational creaming or settling of dispersed droplets in an aqueous emulsion of the present invention.

25 The increase in the viscosity at normal pouring shear rates allows emulsions prepared using the concentrates of the present invention to have properties which are attractive to consumers. Further, aqueous emulsions of the present invention can have viscosities which are

30 relatively consistent between batches. That is,

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particularly with emulsions produced by home dilution, the emulsions are not excessively affected by conditions such as local temperature, concentration variations of concentrate in water, salinity and hardness of dilution water etc. This allows a consistently attractive product to be provided.

It has been found, surprisingly, that the presence of the polymeric structurant in oil emulsions of the present invention also gives improved softening and perfume deposition. The softening feel given to fabric is found to be improved, being silky rather than greasy.

Without being bound by theory, it is believed that aqueous emulsions of the present invention contain droplets of generally spherical shape comprising an outer layer of fabric softening component in lamellar liquid crystalline phase and a liquid core of oil and perfume. It is believed that this structure contributes to the good deposition of perfume onto the fabric.

As will be discussed further below, the cationic fabric softening compound in the concentrates of the present invention may be suspended, dispersed or dissolved in the oil.

Without being bound by theory, when the fabric softening component is suspended or dispersed in the oil, the concentrate compositions of the invention can be described as having a physical state wherein a network of solid

crystallites of controlled strength is formed to contain the liquid phase and the structurant. The strength of the solid network is controlled such that concentrate composition does not undergo gravitational sedimentation under quiescent conditions but flows under agitation and stirring.

Further discussion on the properties of inorganic solids suspended in an oil are given in "Electrostatic Stabilisation of Suspensions in Non-Aqueous Media" PH.C van der Hoeven, University of Wageningen Thesis (1991), Chapter 2.

It is preferred that, when added to water, the emulsified product has an oil droplet size ( $D_{43}$  volume average droplet size) of under  $5\mu\text{m}$  and more preferably under  $3\mu\text{m}$ . Droplet size ( $D_{10}$ ) is typically in the range  $0.2\mu\text{m}$ - $50\mu\text{m}$ .

Stability of the emulsions according to the present invention can be measured by studying the creaming level, for example as discussed below.

#### Polymeric Structurant

As noted above, the water-soluble polymeric structurant is a material which will increase viscosity of an aqueous emulsion of the present invention in water at zero shear rate and at normal pouring shear rates. Preferably, the structurant will be present in the form of small solid particles in the concentrate composition of the

- 10 -

invention. The particles may typically be of size in the range 2-100 micrometres depending on their source. Particle size may be measured by light microscopy. Preferably, the structurant is present as microcrystals.

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Upon dilution with water, the polymeric structurant of the invention becomes fully or partially gelatinised.

This is in contrast to known compositions containing structurants in which the structurant is usually already present in hydrated form, being in the form of a gel or solution, which is post- or pre-dosed into the emulsion. The preferred prior art order of addition of polymers, for examples as set out in EP-A-0596580 in the name of Avebe is to add a concentrated solution of water-soluble polymer after an aqueous emulsion has been prepared.

15

The term "water-soluble" indicates that the polymeric structurant, gelatinises in water at a characteristic temperature range at concentrations of interest, i.e. around 0.1-2% by weight. Preferably, the polymeric structurant when mixed with water does not form a separate phase.

20

The structurant preferably does not have substantial surfactant properties. The structurant is preferably selected from nonionically or cationically modified natural polymers such as protein or plant derived polymers, particularly polysaccharide compositions such as starch. As starting material for the manufacture of

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cationic starch to be used with the present invention, any type of starch can be used such as potato starch, corn starch, wheat starch, tapioca starch or pea starch. The degree of substitution of the cationic starch to be used in accordance with the present invention is preferably in the range 0.005-1 preferably between 0.01 and 0.05. Particularly preferred are forms of starch that have an amylopectin content of more than 95% by weight based on solids.

Suitable methods of manufacturing structurant polymers suitable for use in the present invention are set forth in EP-A-0596580 in the name of Avebe.

Other naturally obtained polymers include cellulose compounds, which may be modified by substituting them with alkyl groups, such as hydrophillically modified hydroxy ethyl cellulose, carboxy methyl cellulose etc. Naturally derived gums such as galactomannan gum or guar gum can be used. Protein derived polymers may include gelatin.

The polymeric structurant used may depend upon the eventual use for which the concentrate is intended. As explained below, the concentrates of the present invention may be used in an industrial or domestic context. Further, the behaviour of some of the concentrates may depend upon the local temperature, hardness of water etc. Cationically modified potato starch such as Solvitose BPN and cationically modified

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Galactomannan, 2-hydroxypropyl trimethyl ammonium chloride ether (Jaguar C-13-S, C-14-S, C-15, C-17 or C-162 - Trade Mark), guar gum or 2 hydroxy-3-(trimethylammonium) propyl ether chloride (HICARE 1000, Registered Trade Mark) and Polygel (Trade Mark) K100 and K200 are preferred for home dilution purposes.

As will be described further below the concentrate can be diluted with water under factory conditions.

Concentrates suitable for dilution under factory suitably comprise cationic potato starches such as SOFTGEL BDA (Trade Mark), SOFTGEL BD (Trade Mark), AMYLOFAX HS (Trade Mark), RAISAMYL 125 (Trade Mark), RAISAMYL 135 (Trade Mark), RAISAMYL 145 (Trade Mark) and synthetic cationic polyacrylic POLYGEL (Trade Mark) K Series, K100 and K200 (3V Sigma).

The amount of polymeric structurant included in the concentrate of the present invention depends upon the desired concentration of polymeric structurant in emulsions of the present invention.

Typically, relatively small quantities of polymeric structurant are required in the emulsions according to the present invention to give the desired viscosities. The quantity in the aqueous emulsion is preferably in the range 0.05-2% by weight, more preferably 0.1-1 wt%, most preferably 0.1-0.5% by weight. The concentration of polymeric structurant in the concentrate is suitably in

the range 1-10% by weight, more preferably 2-5%, most preferably 3-4% by weight.

Without wishing to be bound by theory, it is believed  
5 that the water-soluble polymeric structurant is present in the oil as a suspended phase.

### The Oil

10 The compositions of the present invention comprise at least one oil. The oil may act as a lubricating oil. The oil may be a mineral oil, an ester oil or a sugar ester oil. Some natural oils, such as vegetable oils may be included if appropriate.

15

Fatty alcohols, for example as defined as coactives in EP-A-0394133 are not suitable for use as oils in the present invention. It is preferred that the oils used in the present invention are liquid, whereas the preferred  
20 fatty alcohols of EP-A-0394133 are soft solids.

Preferably, the ester oils are esters of straight chain or branch chain, saturated or unsaturated carboxylic acids.

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Preferably, oils used in the present invention are hydrophobic. Further, it is preferred that they either comprise sugar ester oils or an oil with substantially no surface activity. Fatty alcohols as defined in EP-A-

0349133 are not substantially hydrophobic and are surface active.

It is preferred if the oil is an ester oil, sugar ester  
5 oil or mineral oil. Suitable oils include those in the Sirius range of mineral oils (ex Silkolene).

Suitable ester oils include the saturated ester oils (ex Unichema) and the unsaturated sugar ester oils (ex  
10 Mitsubishi Kagaku).

It is preferred that the ester oils of the invention are hydrophobic in nature. It is further preferred if the ester oil is saturated (hardened) in nature, unless it is  
15 a sugar ester oil or a plant derivative, for which unsaturation is preferred.

Suitable ester oils are the fatty ester of a mono or polyhydric alcohol having from 1 to about 24 carbon atoms  
20 in the hydrocarbon chain and mono or polycarboxylic acids having from 1 to about 24 carbon atoms in the hydrocarbon chain with the proviso that the total number of carbon atoms in the ester oil is equal to or greater than 16 and that at least one of the hydrocarbon radicals in the  
25 ester oil has 12 or more carbon atoms.

Ester oils most suitable for use in the present invention are the PRIOLUBES from Unichema. In particular, PRIOLUBE 1407, PRIOLUBE 1447, PRIOLUBE 1415, PRIOLUBE 1446,  
30 PRIOLUBE 1427, PRIOLUBE 1445, PRIOLUBE 2045, PRIOLUBE



3988, PRIOLUBE 3987, PRIOLUBE 2091, ESTOL 1545, and ESTOL 1527 are advantageously employed. Of these ESTOL 1545, which is a 2-ethyl hexyl stearate are particularly useful.

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Suitable mineral oils include Esso Marcol technical grade range of oils and particularly preferred is the Silkolene medicinal Sirius range (e.g. M40, M70 and M180).

- 10 The molecular weight of the mineral oil is typically within the range 150 to 400.

It is preferred if the density of the mineral oil is from 0.80 to 0.90 g/cm<sup>3</sup>, more preferably from 0.83 to 0.88

15 g/cm<sup>3</sup>.

- It is preferred if the viscosity of the ester oil or mineral oil is from 2 mPa.s to 400 mPa.s at a temperature of 25°C, more preferably a viscosity from 2 to 150 mPa.s, 20 most preferably a viscosity from 10 to 100 mPa.s.

- It is preferred if the viscosity of the sugar ester oil is above 50,000 mPas, preferably 5,000 to 20,000 mPas, most preferably 6,000 to 20,000 mPas. All viscosities 25 are measured at 25°C.

It is further preferred if the refractive index of the oil is from 1.445 to 1.490, more preferred from 1.460 to 1.485.

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The level of oil in the emulsion is preferably in the range 1-15 %wt. The level of oil in the fabric conditioner concentrate is preferably from 20 to 78 wt% of the composition, most preferably from 50 to 70 wt%.

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The Cationic Fabric Softening Compound

The compositions of the present invention comprise at least one fabric softening compound.

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The fabric softening compound is preferably a cationic fabric softening compound.

Preferably, the oil used in the present invention will, when combined with a suitable deposition aid such as conventional cationic fabric softening compounds, have fabric softening properties.

15

The fabric softening compound is more preferably a quaternary ammonium material comprising a polar head group and two alkyl or alkenyl chains.

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Particularly preferably, the fabric softening compound of the invention has two long chain alkyl or alkenyl chains with an average chain length greater than  $C_{14}$ , more preferably each chain has an average chain length greater than  $C_{14}$ , more preferably at least 50% of each long chain alkyl or alkenyl group has a chain length of  $C_{18}$ .

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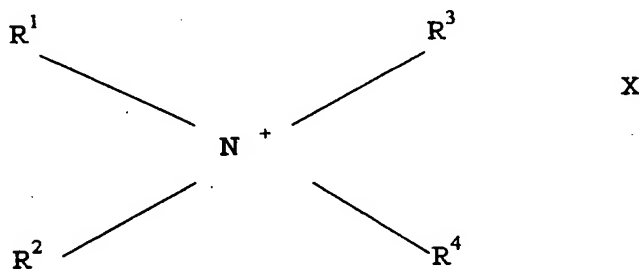
It is preferred if the long chain alkyl or alkenyl groups of the fabric softening compound are predominantly linear.

- 5 It is highly preferred if the fabric softening compounds of the invention are substantially water-insoluble. Substantially insoluble fabric softening compounds in the context of this invention are defined as fabric softening compounds having a solubility less than  $1 \times 10^{-3}$  wt% in  
10 demineralised water at 20°C, preferably the fabric softening compounds have a solubility less than  $1 \times 10^{-4}$ , most preferably the fabric softening compounds have a solubility at 20°C in demineralised water from  $1 \times 10^{-3}$  to  $1 \times 10^{-6}$ .

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Well known species of substantially water-insoluble quaternary ammonium compounds having the formula:

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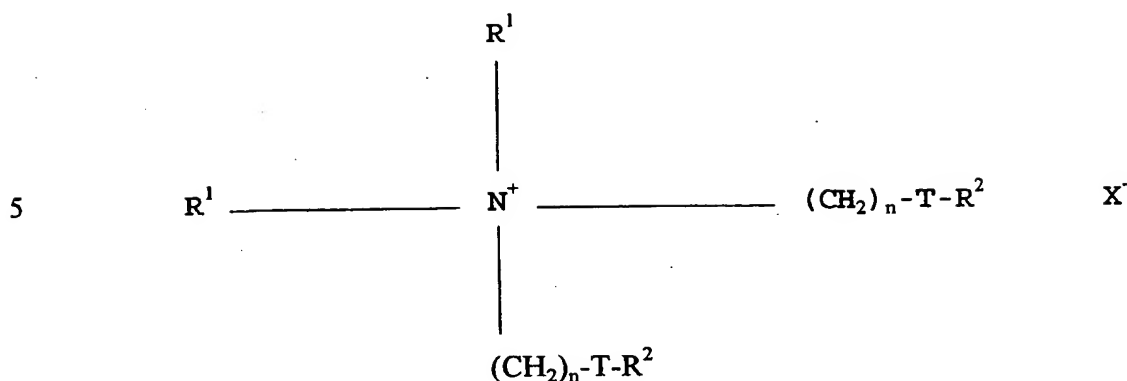
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- wherein  $R^1$  and  $R^2$  represent hydrocarbyl groups having from 12 to 24 carbon atoms;  $R^3$  and  $R^4$  represent hydrocarbyl groups containing 1 to 4 carbon atoms; and X is an anion, preferably selected from halide, methyl  
30 sulphate and ethyl sulphate radicals are preferred.

Representative examples of these quaternary softeners include di(tallow alkyl) dimethyl ammonium methyl sulphate; dihexadecyl dimethyl ammonium chloride; 5 di(hydrogenated tallow alkyl) dimethyl ammonium chloride; dioctadecyl dimethyl ammonium chloride; di(hydrogenated tallow alkyl) dimethyl ammonium methyl sulphate; dihexadecyl diethyl ammonium chloride; di(coconut alkyl) dimethyl ammonium chloride, ditallow alkyl dimethyl 10 ammonium chloride and di(hydrogenated tallow alkyl) dimethyl ammonium chloride (Arquad 2HT Trade Mark).

Other preferred softeners contain esters or amide links, for example those available under the trade names 15 Accosoft 580, Varisoft 222, and Stepantex.

Particularly preferred fabric softening compounds are water-insoluble quaternary ammonium materials which comprise a compound having two C<sub>12-18</sub> alkyl or alkenyl 20 groups connected to the molecule via at least one ester link. It is more preferred if the quaternary ammonium material has two ester links present. The preferred ester-linked quaternary ammonium material for use in the invention can be represented by the formula:

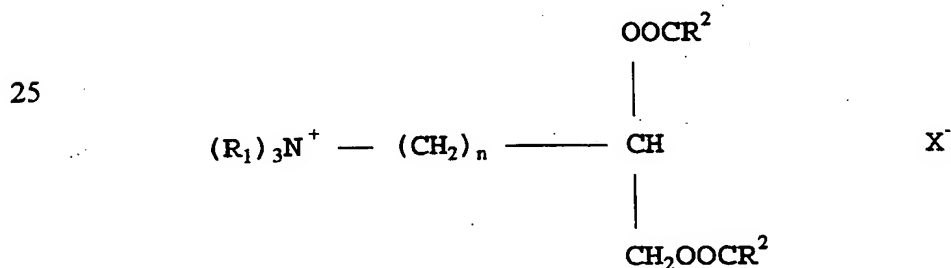


10 wherein each  $R^1$  group is independently selected from  $C_{1-4}$  alkyl, hydroxyalkyl or  $C_{2-4}$  alkenyl groups; and wherein each  $R^2$  group is independently selected from  $C_{8-28}$  alkyl or alkenyl groups;

15 T is  $\begin{array}{c} O \\ || \\ -C-O- \end{array}$  or  $\begin{array}{c} O \\ || \\ -C-O- \end{array}$ ;  $X^-$  is any suitable anion and  $n$  is an integer from 0-5. Particularly preferred is di(ethyl ester) dimethyl ammonium chloride (DEEDMAC).

20

A second preferred type of quaternary ammonium material can be separated by the formula:



30 wherein  $R_1$ ,  $n$ ,  $X^-$  and  $R_2$  are as defined above.

It is advantageous for environmental reasons if the quaternary ammonium material is biologically degradable.

5 Preferred materials of this class such as 1,2 bis[hardened] tallow oxyloxy]-3-trimethylammonium propane chloride and their methods of preparation are, for example, described in US 4 137 180 (Lever Brothers). Preferably these materials comprise small amounts of the corresponding monoester as  
10 described in US 4 137 180 for example 1-hardened tallowoxyloxy-2-hydroxy trimethylammonium propane chloride.

The fabric softening agent may also be a polyol ester quat (PEQ) as described in EP 0 638 639 (Akzo).

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It is preferred that the weight ratio of softening compound to oil is less than 5:1, suitably less than 3:1, preferably less than 1:1. Preferably, the weight ratio of softening compound to oil is greater than 1:10, preferably greater  
20 than 1:3.

25

The level of softening compound in the concentrate is preferably from 3 wt% to 60 wt% of the total composition, more preferably from 10 wt% to 40 wt%. Preferably, dilute emulsions produced according to the present invention contain in the range 0.5-5% by weight cationic active fabric softening compound, more preferably 1-3% by weight, most preferably 1.0-2% by weight.

Without wishing to be bound by theory, where the fabric softening compound is suspended in the oil, it is believed that the fabric softening compound is present in the oil as crystals.

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### Solvent

It is preferred that the composition contains less than 25 wt% of the total composition of organic solvent, more preferably less than 20 wt%, most preferably less than 10 wt%.

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It is especially preferred that the solvents are non-aqueous. In any case level of water must be kept below 10% of the total composition.

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As noted above, the cationic fabric softening compound in the concentrate of the present invention may be dissolved, suspended or dispersed in the oil.

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A cationic fabric softening compound may be soluble in oil if, for example, it comprises carbon chains derived from soft tallow, i.e. tallow having a high iodine.

Alternatively, the cationic fabric softening compound may be made soluble in the oil by inclusion of a suitable solvent. Solvents which may be used include ethanol, propanol, isopropanol, ethylene glycol, 1,2 propylene glycol, 1,3-propylene glycol and glycerol. Suitable solvents preferably have low flash point, for example IPA (isopropyl alcohol).

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For the compositions produced by the melt process, as herein below described, it is preferred that organic solvents are included in the compositions. It is preferred that less than half of the amount of any solvent present is flammable solvent (i.e. has a flash point of less than 25°C). The major proportion of the solvent should most preferably be a non-flammable solvent (i.e. have a flashpoint of higher than 25°C). Suitable examples include propylene glycol and especially hexylene glycol and butyl digol for reasons of viscosity and appearance of the melt. A mixture of solvents may provide advantageous results, especially with respect to viscosity. In some compositions solvent may be present as a result of being a component of an ingredient of the composition.

It is believed that the choice of the type of any solvent present in the compositions of the present invention help to control the size of the crystals of the fabric softening compound, or to make the fabric softening compound soluble in the oil.

#### Perfume

The compositions of the present invention and the method of the present invention are particularly suitable for giving enhanced perfume deposition onto fabric during rinse conditioning. Preferably therefore the concentrate according to the present invention contains perfume suitably at a level in the range 1-10% by weight,



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preferably 3-6% by weight. Suitable perfume compounds may be selected by the person skilled in the art.

Preferably, the perfume is substantially hydrophobic.

- 5 Preferably, the perfume is soluble in oil.

#### Crystal Growth Inhibitor

- 10 The concentrate or emulsions of the present invention may include a crystal growth inhibitor. It is preferred that the crystal growth inhibitor forms part of the concentrate, particularly for non-batch dilution by the consumer at home.

- 15 The crystal growth inhibitors are compounds that have highly polarisable hydrophilic groups.

- Particularly preferred crystal growth modifiers are organic acids of alkyl chain length  $C_{13}$  or less and  
20 nonionic surfactants having an average alkyl chain length between  $C_{13}$  and  $C_{22}$  and from 10 to 30 ethoxylate groups.

- Particularly preferred crystal growth inhibitors are tallow and coco nonionic surfactants having from 15 to 22 ethoxylate groups, organic acids such as lactic acid  
25 (which contains about 20% linear polymeric self-esterified esters), stearic acid, and hardened or unhardened tallow or coco fatty acids. Mixtures of crystal growth inhibitors may also be used.

The crystal growth inhibitor or mixtures thereof should preferably be present at a level of from 1 wt% to 20 wt% of the composition, more preferably the crystal growth inhibitor should be present at a level from 2 wt% to 11 wt%.

#### Co-Emulsifier

It is preferred that the compositions comprise a co-emulsifier for rapid dispersion of the composition when it is added to water. The co-emulsifier may comprise nonionic surfactant.

If a nonionic ethoxylated surfactant is used as the crystal growth inhibitor then this will function as both the inhibitor and as a co-emulsifier to provide good dispersion.

The preferred co-emulsifiers are C<sub>8</sub>-C<sub>22</sub> alcohol alkoxyates with an average of 3 to 10 alkoxyate groups, preferably 5 to 7 alkoxyate groups. Ethoxyates are the preferred alkoxyates although mixed ethoxyates/propoxyates or propoxyates may also be used.

#### Composition pH

The emulsions of the invention when dispersed in water at use concentration preferably have a pH of more than 1.5, more preferably less than 5.

### Product Form

- Concentrates of the present invention are preferably in the form of a paste or high viscosity liquid. The concentrate may be added to water by the consumer or at a factory, to form an emulsion which can then be stored for later use by addition to the rinse liquor.
- 10 The consistency of concentrate compositions produced by the cold grinding method, discussed below, depends upon the melting point of the cationic fabric softener and varies between a liquid dispersion and a paste, whereas products of the hot melt method, discussed below, can have the consistency of a gel if cooled naturally or a pourable
- 15 paste if cooled while agitating.

### Other Ingredients

- 20 The concentrates or emulsions of the invention can also contain one or more optional ingredients, selected from pH buffering agents, perfume carriers, fluorescers, colorants, hydrotropes, antifoaming agents, antiredeposition agents, enzymes, optical brightening agents, opacifiers, anti-
- 25 shrinking agents, anti-wrinkle agents, anti-spotting agents, germicides, fungicides, anti-corrosion agents, drape imparting agents, antistatic agents and ironing aids.

### Method of Production of Concentrate

The concentrate compositions according to the present invention may be produced according to any suitable method. Two methods are particularly preferred, namely the melt process and the cold grinding (milling) process. The  
5 polymeric structurant will generally remain solid in both methods of production.

A melt as referred to herein is a liquid mixture of two or more substances, at least one of which would solidify on  
10 cooling to ambient temperature. The melt may contain dispersed non-liquid material, such as polymeric structurant. In our melts, typically one or two of the components solidify on cooling (usually fabric softening compound, nonionic surfactant and fatty acids, if present).

15

In the melt process the fabric softening compound is heated until it is mobile, preferably liquid, followed by addition to an oil phase (which may contain a perfume) to produce a melt. The polymeric structurant is added to this melt while  
20 the melt is still warm and liquid, the structurant being present as dispersed solid. The additional components of the composition may be incorporated into the composition via a mobile fabric conditioning compound or via the oil, or they may be added after the fabric conditioning compound and  
25 the oil have been mixed together. Typically the melt is formed at a temperature of at least 35°C, preferably of at least 40°C, e.g. at a temperature of 45°C to 70°C. Alternatively, the fabric softening compound and nonionic surfactant(s) are mixed with the oil and then heated to form

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a liquid to which structurant may be added. Perfume may be added upon cooling to a lower temperature.

It is especially advantageous if the softening compound and the oil are heated together to form a melt. Perfume may also form part of the melt.

The melt can cool to a room temperature naturally or while being stirred mechanically. In this manner, the final viscosity of the melt can be further controlled.

Alternatively, the concentrate compositions of the present invention may be produced by a cold grinding (milling) method wherein the fabric softening compound, polymeric structurant and the oil are mixed together, at ambient temperature typically at high shear rates, without the fabric softening composition being heated prior to mixing.

#### Method of Production of Emulsion

20

According to another aspect of the invention, the concentrate compositions of the present invention can be used to prepare an aqueous emulsion by diluting the concentrate composition with water. Alternatively and in another aspect of the present invention, a concentrate composition comprising a fabric softening component mixed with oil may be diluted to form an emulsion using a solution of water-soluble polymeric structurant, which solution comprises water-soluble polymeric structurant at the desired final concentration of the aqueous emulsion.

Preferably, the concentrate is diluted with ambient or hot water. Preferably the temperature of the dilution water is in the range ambient-100°C, preferably 40-70°C, more preferably 50-60°C for batch dilution. Where the  
5 concentrate is in the form of a paste or wax, it may be heated to become a liquid before mixing with water. Mixing of the concentrate with water may take place over a time period of 2-30 minutes, depending on the scale of the mixing process, the equipment used etc. The concentrate  
10 and water may be agitated in any suitable way, for example stirring or shaking.

Where an emulsion is produced by diluting a fabric conditioning concentrate according to the present invention  
15 with water, it is preferred that the polymer is present in the concentrate in substantially solid form. Preferably, it has not been gelatinised by mixing with water before being introduced into the concentrate. Accordingly, in this aspect of the invention, gelatinisation and  
20 emulsification can occur simultaneously. This has been found to give surprising advantages including the ability to control the viscosity and droplet size of the emulsion.

The viscosity of emulsions produced according to the  
25 present invention may depend upon the conditions under which the emulsion is produced by dilution of fabric conditioning concentrate. Further, when producing an emulsion according to the present invention, there is a danger that an undesirable flocculation of the droplets

will occur leading to a creaming and unattractive composition.

The person skilled in the art will be able to avoid such  
5 conditions by trial and error.

However, according to a preferred aspect of the invention,  
the following procedure may be used to determine the  
optimum conditions for production of emulsion according to  
10 the present invention.

For a given polymeric structurant, experiments may be  
conducted to gelatinise the polymer in water under  
controlled conditions of temperature, shear rate and mixing  
15 time, whilst measuring the development of viscosity during  
the mixing process. The optimum shear rate, temperature  
and mixing time for obtaining maximum polymer viscosity for  
a given concentration of polymer in the final emulsion can  
be determined (called 'gelatinisation studies').

20 Viscosity/shear profile measurements can be made using a  
Carri-Med (Trade Mark) controlled stress viscometer with  
cone and plate. The inventor has found that these optimum  
conditions of temperature, shear rate and processing time  
can be applied directly to a batch process for diluting  
25 according to the invention a fabric conditioning  
concentrate.

For the preferred range of polymeric structurants, it is  
found that the optimum batch mixing temperature range is  
30 suitably around 40-70°C, more preferably 50-60°C.

During dilution of concentrate by the process of the present invention, water may be added to the concentrate while mixing (phase inversion route) or concentrate may be added to water. The end result will be substantially the same. It is found that droplet size is normally smaller when water is added to concentrate.

Dilution may be carried out by the household user or in an industrial plant. We have found that inclusion of the polymeric structurant in the concentrate helps the emulsification process and the break-up of droplets such that no extensive processing equipment is required to produce stable emulsions from the concentrates.

#### Examples

The invention will now be illustrated by reference to the following non-limiting examples. Further modifications within the scope of the present invention will be obvious to the skilled person.

All quantities are in parts by weight or weight percent, unless indicated to the contrary.

Comparative examples are illustrated by a letter and Examples of the invention by a number.



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Compositions prepared according to the invention and comparative compositions were subjected to the following tests.

5 In these examples, the term "cationic active" refers to pure cationic fabric softening compound. The term "fabric softener" refers to the commercial product, which is typically 80% pure cationic active and approximately 20% solvent, eg IPA. The term "active softener" is defined  
10 below.

(i) Softness Test

Softening performance is evaluated by adding to 1 ltr of  
15 demineralised water at ambient temperature in a Tergotometer enough product to give 0.1 g of active softener material. The active softener is defined as cationic active or (if oil is present), cationic active plus oil. In this way, the level of active softener was  
20 equal in the rinse liquor for all examples according to the invention. Three pieces of terry towelling (19 cm x 19.5 cm weighing 40 g in total) were added to the Tergotometer pot. The terry towelling was already rinsed in a 0.00045%  
by weight sodium alkyl benzene sulphonate solution to  
25 simulate the anionic of detergent from a main wash. The towels were treated for 5 minutes at 65 rpm, spin-dried to remove excess liquor and line-dried overnight. A panel of  
20 trained people evaluated the towels by comparing against set standards. A low number indicates a greater degree of  
30 softness (2 is very soft and 8 is harsh).

In order to investigate the consistency of the results, the softness measurement was repeated under the same conditions, to give two results for each composition.

- 5 Further, for control, an experiment to measure the softening obtained in a parallel experiment with the same source of water was conducted using COMFORT (Trade Mark), a premium conventional fabric conditioner composition.

10 (ii) Perfume Delivery Evaluation Method

- Perfume delivery was evaluated by rinsing in a Tergotometer three pieces of terry towelling (19 x 19.5 cm weighing 40 g in total) per product in a similar manner to that
- 15 previously described for softening evaluation above. Instead of being line-dried the cloths were immediately assessed for perfume intensity by a trained group of twenty panellists who ranked each cloth on a scale of zero to five corresponding to descriptors ranging from no perfume to
- 20 very strong perfume. Further assessments were made after five hours when the cloths were dry and again after twenty-four hours or longer. The level of product was 0.1 g/l active matter with a perfume level in the rinse liquor of 4.76 mg/l.

25

(iii) Absorbency Test

- Absorbency of fabrics was evaluated by treating terry towelling in a Tergotometer with compositions as described
- 30 for the softening assessment. Strips of fabric were cut to

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11 cm by 3 cm. The strips of treated fabric were held vertically and lowered into a dish containing a 0.02% solution of direct red 81 dye, so that ca. 0.5 cm of the fabric was below the surface of the water. The height to which the liquid rose up the strip was measured at intervals of time for a total of one hour. The average height for each treatment was calculated. Higher values are indicative of better absorbency.

10 (iv) Storage Stability

Emulsions prepared according to the invention or according to the comparative example were stored in cylindrical glass bottles of 60 mm height by 19 mm in diameter at room temperature (approximately 25°C), 37°C and 45°C. Percentage of creaming was calculated according to the following equation:

20 
$$\% \text{ creaming} = \frac{\text{mm separation}}{\text{mm sample height}} \times 100$$

The nature of phase separation was also observed. In particular, it was noted whether or not the separated layer appeared clear (indicating the loss of all droplets into the separated layer) or milky or cloudy (indicating the presence of fine submicron droplets remaining in the aqueous layer).

(v) Viscosity Measurement

Viscosities were measured using a standard Haake viscometer with a NV cup and bob, at a shear rate of  $106 \text{ s}^{-1}$ .

5

Compositions and Methods of Production

Concentrate compositions having the following general compositions were prepared by a cold grinding route. The specific components will be set out further below. In the cold grinding route, fabric softener, oil, perfume and (if included) polymeric structurant were mixed together and then homogenised by a Silverson mixer or overhead Heidolph mixer. As will be explained below, the polymeric structurant may be included in the final emulsion by a different route (route (B)) and be absent from the concentrate.

The oil and structurant used are varied, as will be explained further below.

Unless indicated otherwise, the fabric softener used in these examples is Arquad 2HT which typically contains 80% wt pure cationic active and contains ~ 20% wt isopropyl alcohol (IPA) solvent.

Table 1

Oil:Cationic active wt. ratio	1.4:1	1.8:1	2:1	3:1
Fabric softener (80% cationic active 20% IPA)	41.07	36.26	35.36	27.25
Oil (100% pure)	44.97	50.17	57.19	65.11
Hardened tallow fatty acid	5.19	4.64	-	-
Perfume (100%)	3.90	3.97	4.06	4.17
Structurant Polymer (100% pure) <sup>1</sup>	4.87	4.97	3.38	3.47

<sup>1</sup> unless a different quantity is noted or unless emulsion  
5 is to be prepared by route (B).

The oil:cationic weight ratio is based on the content of pure cationic active.

10 Active:perfume ratio is in most cases fixed at ~ 20.

#### Aqueous Emulsions

Aqueous emulsions were prepared by one of the following  
15 procedures:

(A) by diluting a concentrate comprising perfume, fabric softener and structurant suspended in oil, or

20 (B) by dissolving polymeric structurant in water and diluting a concentrate comprising fabric softener and perfume suspended in oil in the aqueous solution with polymeric structurant.

If necessary, the concentrates were melted first. The water in both routes (A) and (B) was at a temperature in the range 40-70°C. Water and concentrate were mixed for a period of 5-10 minutes. Mixing was carried out in a baffled batch-based apparatus comprising a jacketed vessel linked to a water bath. Stirring was provided by an impeller connected to a overhead Heidolph mixer. A pitch-blade three stage impeller was used, rotating at a rate in the range 300-1,000 rpm.

Aqueous emulsions produced typically contained approximately 5% wt pure active (defined as cationic conditioner plus oil). To prepare them, concentrate was diluted in water or solution of polymeric structurant as set out above, to give the general compositions set out in Table 2. Specific components will be given further below. The fabric softener in each case is Arquad 2HT, as above, unless indicated otherwise. The oil and structurant used are varied, as explained further below.

Table 2

Oil:Cationic active wt. ratio	1.4:1	1.8:1	2:1	3:1
Fabric softener (80% pure cationic active)	2.53	2.19	2.09	1.57
Oil (100% pure)	2.77	3.03	3.33	3.75
Hardened tallow fatty acid	0.32	0.28	-	-
Perfume (100% pure)	0.24	0.24	0.24	0.24
Structurant Polymer <sup>1</sup>	0.30	0.30	0.20	0.20
Water (Demin)	balance	balance	balance	balance

<sup>1</sup> unless indicated to the contrary

### Preparation of Comparative Emulsions

In order to provide a composition without structurant  
5 polymer, a fabric conditioning concentrate was prepared by  
mixing a fabric softener material, perfume and oil by the  
cold grinding route and mixed with water by the method  
described above.

### 10 Results

The following Examples 1-24 and Comparative Examples A-E  
were prepared by preparing the compositions described above  
using the oils and structurants described below. The  
15 compositions, mixing conditions and viscosity of the  
products are shown in Tables 3 to 5.

The route for production of emulsion (explained above) is  
indicated for each table by A or B respectively. The ratio  
20 of oil to pure cationic active in the concentrate is  
indicated for each Table.

### Examples 1 to 11 - Table 3

25 Ratio of oil to cationic active: 2:1. Emulsion  
manufactured by route "B".

Example	Oil	Structurant	Total Batch Size /ml	Mixing Temp °C	Viscosity mPa.s at 106 s <sup>-1</sup>
1	M70	SOFTGEL BD	3000	50	33
2	M180	SOFTGEL BDA	500	50	24
3	M180	AMYLOFAX HS	500	56	29
4	M70	JAGUAR C-13-S	3000	40	36
5	M180	HI CARE 1000	500	50	77
6	M180	GUAR GUM	500	60	20
7	M180	GELATINE <sup>2</sup>	500	60	51
8	M180	SOFTGEL BDA <sup>3</sup>	3000	50	114
9	M180	SOFTGEL BDA	3000	~53	114
10	M70	SOFTGEL BDA <sup>4</sup>	3000	~53	158
11 <sup>5</sup>	ESTOL 1545	SOFTGEL BDA	500	60	17

<sup>2</sup> 0.5 parts by weight, ex BDH

<sup>3</sup> 0.3 parts by weight

5 <sup>4</sup> 0.4 parts by weight

<sup>5</sup> The cationic fabric softener is DEEDMAC, as described on page 18-19, containing about 2% tallow fatty acid impurity, as supplied by Hoechst.

10 M70, M180 are mineral oils ex-SILKOLENE.

SOFTGEL BDA, Amylofax HS and SOFTGEL BD are supplied by AVEBE BA, Holland. HI-CARE 1000 is supplied by Rhone-Poulenc.

15 JAGUAR C-13-S is supplied by Meyhall. Guar gum is supplied by ROHM & HAAS



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Examples 12 to 15 - Table 4

Ratio of oil to cationic active: 1.4:1. Emulsions  
manufactured by route "B".

5

Example	Oil	Structurant	Total Batch size/ml	Mixing Temp °C	Viscosity mPa.s at 106 s <sup>-1</sup>
11	SIRIUS M180	SOFTGEL BDA	3000	60	56
13	SIRIUS M180	SOFTGEL BDA <sup>1</sup>	3000	60	60
14	SIRIUS M180	POLYGEL K100 <sup>2</sup>	200	40	105
15	SIRIUS M180	RAISAMYL 145 <sup>3</sup>	200	65	39

<sup>1</sup> 0.35% SOFTGEL and emulsion contains 4.5% active rather than 5%.

<sup>2</sup> 0.20% POLYGEL K100.

10 <sup>3</sup> 0.20% RAISAMYL and emulsion contains 4.5% active.

Examples 16 and 17 - Table 5

Ratio of oil to cationic active: 1.8:1. Emulsions  
15 manufactured by route "B".

Example	Oil	Structurant	Total Batch size/ml	Mixing Temp °C	Viscosity mPa.s at 106 s <sup>-1</sup>
16	SIRIUS M180	SOFTGEL BDA <sup>1</sup>	3000	60	50
17	SIRIUS M180	SOFTGEL BDA <sup>2</sup>	3000	60	84

<sup>1</sup> Emulsion contains 4.5% active, not 5%.

<sup>2</sup> 0.40% polymer.

20

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Examples 18 to 28 - Table 6

Ratio of oil to cationic active 2:1. Emulsion manufactured by route "A".

Example	Oil	Structurant	Total Batch Size /ml	Mixing Temp /°C	Viscosity/m Pa.s at 106 s <sup>-1</sup>
18	M70	SOFTGEL BD <sup>1</sup>	500	50	77
19	M180	SOFTGEL BDA	3000	50	?
20	M40	SOFTGEL BDA	500	50	85
21	M70	SOFTGEL BD	3000	50	121
22	M70	JAGUAR C-13-S	3000	40	32
23	M180	HI-CARE 1000	500	40	24
24	M70	SOFTGEL BDA	500	52	83
25	M180	SOFTGEL BDA <sup>2</sup>	3000	50	38
26	M180	SOFTGEL BDA	3000	53	38
27	M70	JAGUAR <sup>3</sup> C-13-S	500	50	95
28	M180	AMYLOFAX HS	500	52	39

5

<sup>1</sup> 0.4 parts by weight

<sup>2</sup> 0.15 parts by weight

<sup>3</sup> 0.5 parts by weight

<sup>4</sup> Ryoto sugar ester oil ER-290 (Mitsubishi)

10

Comparative Examples A-D - Table 7

Ratio of oil to cationic active: 2:1

Example	Oil	Structurant	Total Batch Size Vol/ml	Mixing Temp °C	Viscosity mPa.s at 106 s <sup>-1</sup>
A	M40	NONE	500	50	3
B	M70	NONE	500	50	5
C	M180	NONE	500	50	9
D	M180	NONE	3000	~53	5

15

Examples 29 and 30 - Comparative Example E and Table 8

Ratio of oil to cationic active: 3:1

Example	Oil	Structurant	Total Vol/ml	Mixing Temp °C	Viscosity mPa.s at 106 s <sup>-1</sup>
29	M70	SOFTGEL BDA	500	50	34
E	M70	NONE	500	50	12
30	M70	SOFTGEL BD	500	50	101

5 Results - Creaming - Table 9

The compositions of examples above were tested for creaming. The results are set out below. The figures show %wt creamed out.

Example	1 week			4 weeks		
	25°C	37°C	45°C	25°C	37°C	45°C
A	5	25	26	11	33	31
20	0	0	0	0	0	0
B	-	-	-	9	38	51
1	-	-	-	0	12	15
18	-	-	-	-	-	-
4	-	2	5	0	24	39
				CLEAR	CLEAR	CLEAR
10	-	-	-	-	-	-
21	-	-	-	0	0	0
22	-	-	-	-	-	-
23	-	-	-	-	-	-
27	0	0	0	0	0	0
C	-	-	-	-	-	-
D	12	83	85	88	84	86
2	0	0	0	0	0	0
3	0	1	2	0	11	12
5	12	33	36	15	50	56
	CLEAR	CLEAR	CLEAR	CLEAR	CLEAR	CLEAR
6	13	68	41	15	58	67
7	0	92	86	5	89	89
8	0	0	0	0	0	0
9	0	0	0	0	0	0
19	-	-	-	-	-	-
25	-	-	-	-	-	-
26	0	0	0	0	6	18
28	-	-	-	-	-	-
11	-	-	-	-	-	-
12	0	0	0	0	0	0
13	0	0	0	0	0	0
14	0	0	0	0	0	0
15	0	0	0	0	0	8
16	0	0	0	0	0	0
17	0	0	0	0	0	0

- = not measured.

- Comparison of Example 20 with the Comparative Example A
- 5 (having no structurant polymer) clearly shows that excessive creaming occurs with the comparative example but is absent over a substantial period and normal temperature range for an emulsion produced by the present invention.

Comparison of Examples 1, 21 and 27 with Example B shows substantially less creaming for compositions of the present invention having structurant polymer. In Example 4, some creaming did occur, but the product is clear rather than  
 5 turbid, indicating that flocculation has not occurred.

Results - Perfume Intensity - Table 10

The compositions of examples were tested for perfume  
 10 intensity. The results are set out below.

Example	Perfume Intensity				
	0 hours	5 hours	24 hours	48 hours	72 hours
A	-	-	-	-	-
20	3.44	2.40	2.29	-	-
B					
1	2.94	2.43	1.93	-	-
18	-	-	-	-	-
4	-	-	-	-	-
10	-	-	-	-	-
21	-	-	-	-	-
22	-	-	-	-	-
23	3.45	1.56	1.18	-	0.75
27	-	-	-	-	-
D	4.03	2.28	1.25	-	0.89
2	-	-	-	-	-
3	-	-	-	-	-
5	3.97	2.79	2.62	1.81	-
6	-	-	-	-	-
7	3.86	2.64	2.24	1.56	-
8	-	-	-	-	-
9	3.69	2.64	1.98	1.06	-
19	-	-	-	-	-
25	-	-	-	-	-
26	-	-	-	-	-
28	-	-	-	-	-
11	-	-	-	-	-
12 <sup>2</sup>	4.10	2.30	1.50	0.98	-
16 <sup>2</sup>	3.40	2.00	1.60	0.90	-
CONTROL <sup>1</sup>	2.33	0.89	0.33	-	0.75

- = not measured

<sup>1</sup> COMFORT (Trade Mark), a current premium conventional fabric conditioner.

5 <sup>2</sup> Carryover of anionic = 0.001%.

In general, compositions of the present invention provide much higher perfume intensity than the current premium conventional fabric conditioner. Further, compositions  
10 having polymeric structurant have similar perfuming properties to emulsions based on oil based concentrate not having polymeric structurant.

Results - Softness Scores - Table 11

15

The compositions of examples were tested for softness score. The results are set out below.

- 45 -

	Softness Score (CONTROL in brackets)	
A	6.63 (5.75)	-
20	5.13 (5.75)	4.75 (4.00)
B	4.25 (3.75)	4.88 (3.38)
1	3.63 (3.75)	4.75 (3.63)
18	-	-
4	-	-
10	-	-
21	-	-
22	-	-
23	-	-
27	-	-
C	5.00 (4.63)	5.75 (3.38)
D	4.75 (3.75)	-
2	-	-
3	4.75 (5.88)	7.0 (5.75)
5	4.25 (3.88)	-
6	4.25 (3.88)	-
7	-	-
8	-	-
9	-	-
19	-	-
25	-	-
26	-	-
28	-	-
11	4.63 (6.19)	-
1B <sup>2</sup>	4.30 (4.40)	-
2B <sup>2</sup>	4.00 (3.50)	-
4B <sup>2</sup>	4.38 (4.00)	-
1C <sup>2</sup>	3.60 (3.50)	-
2C <sup>2</sup>	4.40 (4.00)	-

- = not measured.

5. Notes<sup>1</sup> COMFORT - Trade Mark.<sup>2</sup> Carryover of anionic = 0.001%

These results measure absolute softening effect and do not  
 10 show the quality of softening. Compositions of the present

invention give similar or better softening compared to the premium conventional fabric softening compositions.

Compositions made according to the present invention have similar softening to compositions not having any polymeric structurant.

#### Examples 23-27

In order to investigate the properties of emulsions produced in a simulated home use situation, the following experiments were conducted.

Fabric conditioning concentrates were manufactured by route (B) described above.

Compositions were then tested as follows. 6.0 g of concentrate was weighed into a bottle and 94 g of tap water added to the bottle. The mixture is shaken for about two minutes. Emulsions are found to form spontaneously. The viscosity and softening results are set out below. The tap water was supplied at 15°C. The compositions had the following general composition.

Oil:cationic active	2:1
fabric softener (80% cationic active) (Arquad 2HT)	35.43
oil (100%) (M180 - ex-SILKOLENE)	53.81
nonionic coco (EO)20 (100%)	5.38
perfume (100%)	5.38
structurant polymer	variable



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In this case, a single measurement of softening performance was made for the concentrates of the invention and for a control (COMFORT, Registered Trade Mark).

5 The results are set out in Table 12.

Solvitose BPN is a cationic potato starch polymer supplied by AVEBE.

10 The level of anionic carryover was 0.001%.

Table 10

Example	Polymer	Parts by weight	Viscosity mPas at 106 s <sup>-1</sup>	Softness score	Viscosity at 106 s <sup>-1</sup> after 4 weeks
31	SOLVITOSE BPN	0.50	83	3.56 (4.5)	99
32	SOLVITOSE BPN	1.0	143	-	109
33	JAGUAR C-13-S	0.50	142	-	141
34	HI-CARE 1000	0.50	97	-	98
35	POLYGEL K100	0.10	65	-	75

15 - = not measured.

Perfume Performance

Example	Perfume Results			
	Wet	5 hrs	24 hrs	48 hrs
31	3.41	2.02	1.49	0.95
CONTROL <sup>1</sup>	2.26	1.00	0.69	0.62

20 <sup>1</sup> COMFORT (Registered Trade Mark, a premium conventional fabric softening composition.)

Absorbency Results

Example	Wicking Height (cm)
CONTROL <sup>1</sup>	3.6
31	9.9
UNTREATED	20.0

5 The absorbency results are a measure of the hydrophobicity given to fabrics treated with the fabric softening composition. The results show that emulsions produced by the present invention have very good re-wetting non-hydrophobising properties so that towels treated with these can be used to dry effectively.

**CLAIMS:**

1. A conditioning concentrate composition comprising a fabric softening compound mixed with oil, wherein the composition contains a water-soluble polymeric structurant.
2. A conditioning concentrate composition according to claim 1, wherein the water-soluble polymeric structurant is present in the form of solid particles.
3. A conditioning concentrate composition according to claim 1 or 2, wherein the water-soluble polymeric structurant has substantially no surfactant properties.
4. A conditioning concentrate composition according to claims 1, 2 or 3, wherein the water-soluble polymeric structurant comprises a nonionically or cationically modified natural polymer.
5. A conditioning concentrate composition according to any of claims 1-4, comprising 1-10% by weight of water-soluble polymeric structurant.
6. A conditioning concentrate composition according to any of claims 1-5, wherein the oil is substantially hydrophobic.
7. A conditioning concentrate composition according to any of claims 1-6, wherein the oil is selected from the group consisting of mineral oils, ester oils, sugar ester oils or mixtures thereof.

8. A conditioning concentrate composition according to any preceding claim, comprising 20-78 wt% of oil.

5 9. A conditioning concentrate composition according to any preceding claim, wherein the fabric softening compound comprises a quaternary ammonium compound.

10 10. A conditioning concentrate composition according to any preceding claim, comprising from 3-60 wt% of fabric softening compound.

11. A conditioning concentrate composition according to any preceding claim further comprising perfume.

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12. A method of manufacturing a conditioning concentrate composition according to any one of claims 1 to 10, comprising mixing a fabric softening compound, an oil and a water-soluble polymeric structurant.

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13. A method of preparing an aqueous emulsion containing a fabric conditioning component, comprising diluting the conditioning concentrate of any one of claims 1 to 10 with water.

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14. A method of preparing an aqueous emulsion containing a fabric conditioning component, comprising dissolving a water-soluble polymeric structurant in water to provide a solution of polymeric structurant of a concentration substantially equal to the desired final concentration of water-soluble polymeric structurant in the emulsion, using

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the solution of polymeric structurant to dilute a conditioning concentrate composition comprising a fabric softening compound mixed with oil.

- 5 15. A method according to claim 13 or 14, wherein the water-soluble polymeric structurant is selected from the group consisting of nonionically or cationically modified natural polymers or mixtures thereof.
- 10 16. A method according to claim 13, 14 or 15 wherein the oil is selected from the group consisting of mineral oils, ester oils or sugar ester oils.
- 15 17. A method according to any one of claims 13 to 16, wherein the fabric softening compound comprises quaternary ammonium compound.
- 20 18. A fabric conditioning composition comprising an aqueous emulsion, the aqueous emulsion comprising water-soluble polymeric structurant, oil and fabric softening compound.
- 25 19. A fabric conditioning composition according to claim 18, wherein the polymeric structurant is selected from the group consisting of nonionically or cationically modified natural polymers or mixtures thereof.
- 30 20. A fabric conditioning composition according to claim 18 or 19, comprising 0.05-2% by weight of water-soluble polymeric structurant.

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21. A fabric conditioning composition according to claim 18, 19 or 20, wherein the oil is selected from the group consisting of mineral oil, ester oil, sugar ester oil and mixtures thereof.

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22. A fabric conditioning composition according to any of claims 18-21, comprising 1-15% by weight of oil.

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23. A fabric conditioning composition according to any of claims 18-22, wherein the fabric softening compound comprises a quaternary ammonium compound.

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24. A fabric conditioning composition according to any of claims 18-23, wherein the fabric softening compound is present at a level of 0.5-5% by weight.

25. A fabric conditioning composition according to any of claims 18-24, having a viscosity in the range of 10-100 mPa.s at 106s<sup>-1</sup>.

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# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP 99/00497

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 6 C11D3/00 C11D3/22 C11D1/835

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 813 862 A (HENKEL KGAA) 29 December 1997 see page 4, line 18 - line 22; claims 1,1; examples	1,9-12, 18
A,P	EP 0 829 531 A (UNILEVER PLC ;UNILEVER NV (NL)) 18 March 1998 cited in the application see claims 1-18; examples	1,6-11, 18
A	US 5 429 754 A (LIN SAMUEL Q ET AL) 4 July 1995 see column 5, line 43 - column 6, line 11; claim 1	1
A	US 5 246 611 A (TRINH TOAN) 21 September 1993 see claims; examples 22,32	1-3

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

\* Special categories of cited documents:

- "A" document defining the general state of the art which is not considered to be of particular relevance
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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 99/00497

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
EP 0813862	A	29-12-1997	DE 19624051 A	18-12-1997
EP 0829531	A	18-03-1998	BR 9704481 A	22-12-1998
US 5429754	A	04-07-1995	CA 2147420 A	04-11-1995
US 5246611	A	21-09-1993	NONE	